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Deliverable Report
D1.7 Performance of final prototype version

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1 Introduction to the project SERPIC

The project *Sustainable Electrochemical Reduction of contaminants of emerging concern and Pathogens in WWTP effluent for Irrigation of Crops – SERPIC* will develop an integral technology, based on a multi-barrier approach, to treat the effluents of wastewater treatment plants (WWTPs) to maximise the reduction of contaminants of emerging concern (CECs). The eight partners of the SERPIC consortium are funded by the European Commission and by six national funding agencies from Norway, Germany, Italy, Spain, Portugal and South Africa. The official starting date of the SERPIC project is 1st September 2021. The project had a duration of 36 months, obtained an extension of 4 months and thus it will end 31st December 2024.

The overall aim of the SERPIC project is to investigate and minimise the spread of CECs and antimicrobial resistant bacteria/antibiotic resistance genes (ARB/ARG) within the water cycle from households and industries to WWTPs effluents, and afterwards via irrigation into the food chain, into soil and groundwater and into river basins, estuaries, coastal areas, and oceans with a focus on additional water sources for food production.

A membrane nanofiltration (NF) technology will be applied to reduce CECs in its permeate stream by at least 90 % while retaining the nutrients. A disinfection using ozone, produced electrochemically, will be added to the stream used for crops irrigation (Route A). The CECs in the polluted concentrate (retentate) stream will be reduced by at least 80 % by light driven electro-chemical oxidation. When discharged into the aquatic system (Route B), it will contribute to the quality improvement of the surface water body.

A prototype treatment plant will be set-up and evaluated for irrigation in long-term tests with the help of agricultural test pots. A review investigation of CECs spread will be performed at four regional target countries in Europe and Africa. It will include a detailed assessment of the individual situation and surrounding conditions. Transfer concepts will be developed to transfer the results of the treatment technology to other regions, especially in low- and middle-income countries.

2 Report summary

In this report, SERPIC results related to the removal of the selected CECs and their concentration in the effluents of the two routes (A and B) are compared with the performance of conventional additional treatments adopted in the case of an agriculture reuse of the treated effluent (depth filtration and disinfection) and CECs content in surface water (rivers) of the four regional target countries. Referring to CECS, it emerges that conventional treatment effluent as well as surface water used for crops irrigation might have higher concentrations with respect those measured in SERPIC effluents. An advanced multi-barrier treatment system, such as that developed by SERPIC project, is more prone to maximize the reduction of a wide range of contaminants from a secondary effluent.

3 Deliverable description as stated in the Project Description

A comparative report with the information from **T1.3**, **T2.9** and **T2.10** about the quality of the SERPIC solution, by a conventional reclaiming process (filtration + UV irradiation) and that used typically by farmers, withdrawn directly from the environment (surface water) will be made. This report will also include information on how irrigation with the three types of water influences the distribution of the target CECs in the irrigated soil and in the crops.

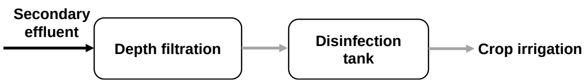
4 Introduction

The recent EU regulation on water reuse (2020/741) identifies different quality classes of water (A-D) which will be destinated to irrigate different types of crops. Water quality classes may be obtained by adopting different treatment trains. The EU regulation reports “indicative technology targets”: secondary treatment, filtration and disinfection. The attention is towards conventional water parameters: *E. coli*, BOD_5, TSS, turbidity, Legionella and intestinal nematodes. The European guidelines to support the regulation on water reuse (EC 2022/C 298/01) pay also attention to the presence of chemical pollutants which can be still present in the treated effluent and may pose adverse effects on the environment and the human health. They are substances characterized by a small molecular size, very different chemical and physical properties, different behaviours during (waste)water treatment and belong to the group of recalcitrant dissolved organic compounds. Their size may be in the order of the nm, smaller than viruses and of the same order of aqueous salts. Conventional depth filtration is not able to retain these compounds as removal mechanisms occurring within the filter medium is mainly size exclusion. The following chemical disinfection can remove some compounds but most of them are not removed. To be able to effectively remove CECs, advanced treatment steps should be integrated in conventional treatment trains. A possible solution is the technology developed by the SERPIC project.

5 Results

For agriculture reuse, the conventional treatment process (Figure 1-A) is based on activated sludge treatment, depth filtration and disinfection, mainly chlorination or UV (Metcalf and Eddy, 2007). As showed in Figure 1-B, the SERPIC prototype plant consists of a membrane nanofiltration step fed by the secondary effluent and producing a permeate, which is subsequently disinfected with ozone (Route A) and a concentrate, that is then oxidized in a photoreactor where electrochemically produced persulfates are added (Route B). Route A effluent is suitable for crop irrigation, Route B effluent for the discharge into rivers.

A.



B.

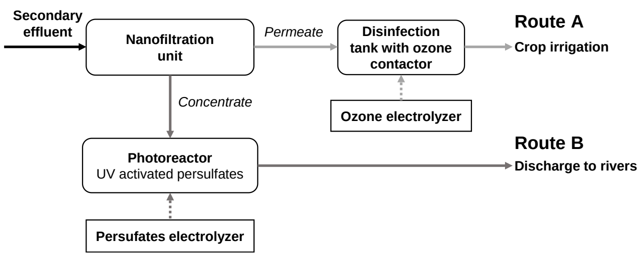


Figure 1. A. Conventional treatment train for agriculture reuse (Metcalf and Eddy, 2007).
B. Advanced treatment chain, developed within SERPIC project.

Table 1 compares organic and microbial CECs removal by the conventional treatments (depth filtration, chlorination and UV radiation) with those obtained by Route A (nanofiltration and ozonation) of SERPIC technology. Organic (diclofenac DIC, iopromide IOP, sulfamethoxazole SMX and venlafaxine VNLX) and microbial (*E.coli* and *sul*1) contaminants are those selected for the performance evaluation in this project (Deliverable D1.1). It is not possible to compare the removal efficiencies for the whole end-of-pipe treatment trains due to lack of literature data. Values of CECs removal in single conventional treatments fed by activated sludge effluent refer to investigations at laboratory, pilot or full scale and real wastewater. Data regarding the removal achieved by depth filtration were not found for IOP, VNLX, *E. coli* and *sul*1 and by chlorination for IOP. In the study by Cibati et al., 2022 the secondary treatment guarantees an effluent of < 1 mg/L of suspended solids as in the permeate of a membrane bioreactor (this corresponds to a conventional activated sludge followed by a depth filtration step).

The SERPIC investigations results refer to an ozone dosage of 2.1 mg O_3/L (which referred to the dissolved organic carbon (DOC) in the influent of 0.81 mg O_3/mg DOC). This ozone dosage was obtained considering that the ozone mass flow rate added to the disinfection unit was 36 mg/h and the influent flow rate (nanofiltration permeate) was in the range between 13L/h and 20 L/h.

The removal of *sul*1 shown in Deliverable D1.4 were here expressed in terms of Log units reduction (by means of equation 1), in order to compare SERPIC results with those found in literature.

$$\text{Log reduction of } sul1 = \log_{10}\left(\frac{C_0}{C_i}\right) \qquad \text{Equation 1}$$

where C_0 and C_i are the gene copy number of *sul*1 respectively in secondary effluent and after the added treatment.

After specific inspection tests carried out by the supplier technicians, it emerged that delivered nanofiltration membranes there was a problem with membrane assembly leading to leaks during the operation, which justified the low removal of *E.coli* (Table 1), much lower than the expected one and the values reported by other investigations (among them Krzeminski et al., 2017).

Table 1: Comparison of CEC removal by the conventional treatment (depth filtration and disinfection) and the SERPIC treatments of Route A, i.e. nanofiltration (NF) and ozonation.

CEC	Treatment	Scale of study	Dosage	Average removal	Reference
DIC	Depth filtration	Pilot		6.8%	Rizzo et al., 2015
	UV radiation	Pilot	127-684 mJ/cm^2	100%	Cibati et al., 2022
		Full	27 mJ/cm^2	31.0%	Paredes et al., 2018
	Chlorination	Full		60.0%	Anumol et al., 2016
	NF_SERPIC	Pilot		90.6%	Deliverable D1.4
	Ozonation_SERPIC	Pilot	2.1 mg/L	51.6%	Deliverable D1.4
IOP	UV radiation	Pilot	127-684 mJ/cm^2	84.0%	Cibati et al., 2022
	NF_SERPIC	Pilot		87.7%	Deliverable D1.4
	Ozonation_SERPIC	Pilot	2.1 mg/L	58.9%	Deliverable D1.4
SMX	Depth filtration	Pilot		28.0%	Mitchell and Ullman 2016
		Full		26.9%	Nakada et al., 2007
	UV radiation	Pilot	112-684 mJ/cm^2	76.0-100%	Cibati et al., 2022
		Full	27 mJ/cm^2	33.0%	Paredes et al., 2018
	Chlorination	Full		48.0%	Anumol et al., 2016
		Full		27.0%	Li and Zhang 2011
		Full		89.6%	Renew and Huang, 2004
	NF_SERPIC	Pilot		84.8%	Deliverable D1.4
	Ozonation_SERPIC	Pilot	2.1 mg/L	52.9%	Deliverable D1.4
VNLX	UV radiation	Pilot	127-684 mJ/cm^2	0-13%	Cibati et al., 2022
	Chlorination	Full	0.2–9 mg/L	71.0%	Golbaz et al., 2023
	NF_SERPIC	Pilot		80.9%	Deliverable D1.4
	Ozonation_SERPIC	Pilot	2.1 mg/L	44.9%	Deliverable D1.4
E. coli	UV radiation	Laboratory	10 mJ/cm^2	90.7%	Wang et al., 2023
	Chlorination	Laboratory	2 mg/L	99.9%	Wang et al., 2023
	NF_SERPIC	Pilot		24.5%	Deliverable D1.4
	Ozonation_SERPIC	Pilot	2.1 mg/L	97.1%	Deliverable D1.4
***sul*1**	UV radiation	Laboratory	249.5 mJ/cm^2	0.38 log	Zhang et al., 2015
		Laboratory	12,477 mJ/cm^2	2.7 log	Zhuang et al., 2015
	Chlorination	Laboratory	30 mg/L	1.2 log	Zhang et al., 2015
		Laboratory	160 mg/L	3.2 log	Zhuang et al., 2015
	NF_SERPIC	Pilot		0.25 log	Deliverable D1.4
	Ozonation_SERPIC	Pilot	2.1 mg/L	1.56 log	Deliverable D1.4

Conventional treatments aim to produce an effluent that meets the reclaimed water quality standards set by European Regulation 2020/741. Currently, legal limits are defined for organic matter, suspended solids, turbidity and microorganisms (*E.coli*, Legionella, intestinal nematodes).

Regarding CECs, still unregulated, depth filtration is not efficient in removing them (Table 1). Disinfection (with UV or chlorine) can achieve different levels of removal depending on the applied dosage. For instance, high removal of DIC, IOP and SMX (> 70%) are possible, by applying a UV dose up to 684 mJ/cm^2 (Cibati et al., 2022), which is about 6 times higher than the typical dose (100 mJ/cm^2) required to achieve total coliform disinfection in activated sludge effluent for a direct reuse (Metcalf & Eddy 2007). The common dosages applied at full-scale plants (around 50 mJ/cm^2) for release in the environment is not sufficient to reduce the concentration of CECs.

If CECs removal is requested in a reuse project, SERPIC technology may be a valid alternative. In fact, it showed to fulfil the European Regulation 2020/741 quality standards (Table 6 of Deliverable 1.4), and it also demonstrated to be able to achieve high CECs removal. The nanofiltration unit reduced their concentration by at least 80 % (DIC < IOP < SMX < VNLX), and the following ozonation completes the removal of *E. coli* and *sul*1 to a few units in 100 mL (Table 2). The literature confirms the SERPIC results: Nanofiltration typically removes CECs with molecular weight in the range 300–1000 g/mol (as DIC 296.1 g/mol, IOP 791.1 g/mol, SMX 253.3 g/mol and VNLX 277.4 g/mol) and ozonation is effective in oxidizing the small recalcitrant compounds still present and in completing the inactivation of ARBs and in (partially) removing ARGs (Rizzo et al., 2020). The SERPIC project includes a Route B for the treatment of nanofiltration concentrate aiming to overcome the problem of CECs accumulation in the concentrate of membrane processes.

The SERPIC prototype plant produces two effluents which can be directly re-used for irrigation (Route A) or released into the environment (Route B). As the farmers typically withdraw water for irrigation from surface water (SW) bodies, concentration of selected CECs (DIC, IOP, SMX and VNLX) in surface water in the four regional target countries (Italy, Portugal, Spain and South Africa) was collected from literature for a comparison. Table 2 reports the range of variability of the collected data in the main references as well as the CECs concentrations found in Route A and Route B effluents of the SERPIC technology. Regarding literature data, no concentration data were found for IOP in Italian and Portuguese SW and *sul*1 in SW in South Africa.

More in details, the box plots of Figures 1 and 2 show the distribution of the concentrations of the selected organic and microbial CECs in the surface water of the four countries and in the two SERPIC water streams. The box plots show the first quartile (25 % percentile), the median and the third quartile (75 % percentile) of the data set. The whiskers are the minimum and the maximum concentrations of the data set.

Conventional WWTPs remove organic CECs in wastewater only partially; as a consequence, CECs are still present in the effluent discharged into surface water. As shown in Table 2, in surface water the organic CECs concentration is in the range 0.2 ng/L – $2x10^4$ ng/L, *E. coli* content is in the range 10^1 CFU/100 mL – $3x10^4$ CFU/100 mL and *sul*1 concentration varies from 7 n°copies/mL to $2x10^6$ n°copies/mL. Using this type of water for irrigation can increase the potential health risks of crops, as CECs have been shown to accumulate in some edible part of the plants (Ben Mordechay et al., 2022). It is important to note that most of the data shown in Table 2 refer to surface water samples collected near a WWTP, whereas water for irrigation is normally withdrawn a few kilometres far from the point of discharge of a WWTP. The fate of CECs in natural environments may change in this stretch due to natural attenuation as well as degradation and other transformation processes.

In both Routes, SERPIC prototype plant is able to reduce organic and microbial CECs concentration below the limit of detection of the instrument (see Table 2), except for VNLX. Excluding VNLX, the discharge of Route B effluent in surface water will not increase its CEC content. Route A effluent can be used for crop irrigation due to the very low CEC concentration. Attention must be paid to nutrients concentrations, as they may not be adequate for crop requirements (Yalin et al., 2023).

Table 2: Comparison of CEC concentrations in surface water (SW) in the four regional target countries (Italy, Portugal, Spain and South Africa) and in the two SERPIC effluent water streams (Route A and Route B). LOD: limit of detection.

CEC	Source	Concentration (range)	Reference
DIC	Italy SW	2.82 – 1,537 ng/L	Castiglioni et al., 2020
	Portugal SW	1.40 – 4,806 ng/L	de Jesus Gaffney et al., 2015; Palma et al., 2021
	Spain SW	4.08 – 319 ng/L	Čelić et al., 2019; Silva et al., 2011
	South Africa SW	5.64 – 20,800 ng/L	Madikizela and Chimuka, 2016; Mhuka et al., 2020
	Route A effluent	45.0 ng/L (< LOD)	
	Route B effluent	45.0 ng/L (< LOD)	
IOP	Spain SW	14.0 – 5,125 ng/L	Acuña et al., 2015; Gros et al., 2012
	South Africa SW	247 – 814 ng/L	Archer et al., 2017
	Route A effluent	140 ng/L (< LOD)	
	Route B effluent	140 ng/L (< LOD)	
SMX	Italy SW	0.85 – 421 ng/L	Cardini et al., 2021; Mandaric et al., 2017
	Portugal SW	1.70 – 230 ng/L	Palma et al., 2021; Silva et al., 2021
	Spain SW	1.39 – 99.0 ng/L	Čelić et al., 2019; Mandaric et al., 2018
	South Africa SW	3.30 – 10,568 ng/L	Segura et al., 2015
	Route A effluent	20.0 ng/L (< LOD)	
	Route B effluent	20.0 ng/L (< LOD)	
VNLX	Italy SW	1.60 – 197 ng/L	Mandaric et al., 2017
	Portugal SW	0.16 – 641 ng/L	Fernandes et al., 2020; Reis-Santos et al., 2018
	Spain SW	0.45 – 349 ng/L	Čelić et al., 2019; Mandaric et al., 2018
	South Africa SW	0.17 – 107 ng/L	Archer et al., 2017; Mhuka et al., 2020
	Route A effluent	40.0 (< LOD) – 99.0 ng/L	
	Route B effluent	339 – 455 ng/L	
E. coli	Italy SW	151 – 33,700 CFU/100 mL	Manini et al., 2022
	Portugal SW	400 – 11,333 CFU/100 mL	Bessa et al., 2014
	Spain SW	2,221 – 8,775 CFU/100 mL	Jurado et al., 2019
	South Africa SW	79.2 – 29,600 CFU/100 mL	Edokpayi et al., 2015
	Route A effluent	1.0 (< LOD) - 13.0 CFU/100 mL	
	Route B effluent	1.0 CFU/100 mL (< LOD)	
***sul*1**	Italy SW	18.2 – $1.58x10^6$ n°copies/mL	Pantanella et al., 2020;
	Portugal SW	480 – $1.40x10^5$ n°copies/mL	Cacace et al., 2019
	Spain SW	7.19 – $4.79x10^5$ n°copies/mL	Calero-Cáceres et al., 2017
	Route A effluent	1.0 (< LOD) – 2.35 n°copies/mL	
	Route B effluent	1.0 (< LOD) – 39.4 n°copies/mL	

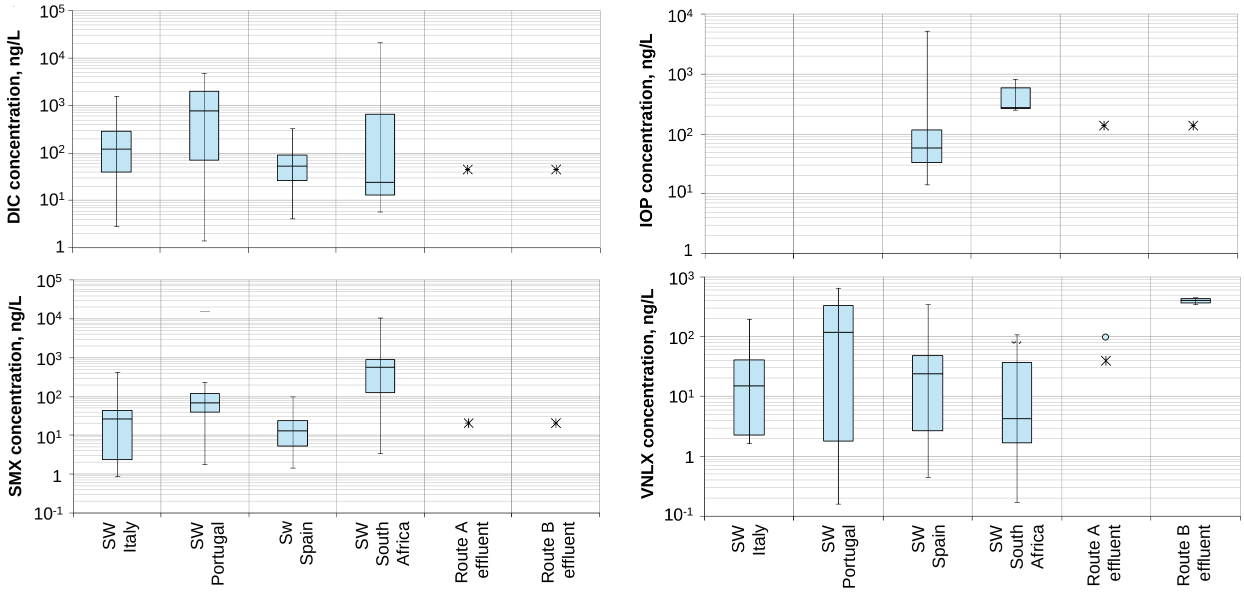


Figure 1: Box plots representing the concentrations of the selected organic CECs (DIC, IOP, SMX and VNLX) in surface water (SW) in the four regional target countries (Italy, Portugal, Spain and South Africa) and in the two effluents of SERPIC prototype plant (Route A and Route B). For SERPIC results, stars (∗) are the concentrations below the limit of detection (LOD), while circles (o) represent the value above the LOD. See Table 2 for references.

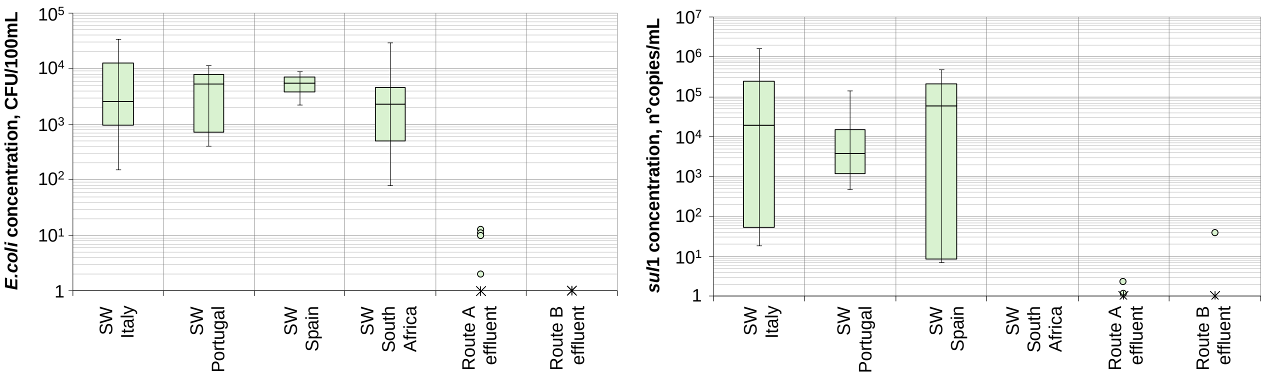


Figure 2: Box plots representing the concentrations of the selected microbial CECs (*E.coli* and *sul*1) in surface water (SW) in the four regional target countries (Italy, Portugal, Spain and South Africa) and in the two SERPIC product water streams (Route A and Route B). For SERPIC results, stars (∗) are the concentrations below the limit of detection (LOD), while circles (o) represent the value above the LOD. See Table 2 for references.

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